

DEVELOPMENT OF HEAT-SEALABLE SEWING THREAD

PHASE I

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| <p>Developmental CP fabrics contain a waterproof breathable membrane (Gore-Tex)[®] barrier which is laminated to the underside of an outer shell fabric. This barrier provides liquid water resistance and some degree of agent resistance. During sewing this barrier becomes perforated, inviting penetration by liquid and vaporous agents, thereby posing a severe hazard to the soldier. This problem is presently handled by using a hot melt tape to seal the thread bundles and holes generated by sewing.</p> <p>The purpose of this program was to screen and evaluate hot melt coatings for thread which could heat-seal the holes made during sewing.</p> <p>Commercially available hot melt coatings investigated included: polyesters, polyamides, polyurethanes, vinyls, and ethylene copolymers.</p> | | | | | |
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19. Abstract - continued

Quantitative peel strength data for the hot melts versus polyester, nylon and Gore-Tex® were developed. The best hot melts were coated onto nylon thread, which was then sewn into nylon Taslan*/Gore-Tex/tricotknit cloth. The sewn cloth samples were tested for hydrostatic resistance.

Based on the results of the hydrostatic resistance tests, the adhesive chosen for pilot plant coating and machine sewing was Michem® 4990, an ethylene/acrylic acid copolymer based material supplied by Michelman/Dow Chemical Company.

In summary, this Phase I effort demonstrated the concept of coating thread with a hot melt resin and briefly heating the sewn seam of an outer shell fabric to effect a seal, thereby preventing hydrostatic leakage. Several hot melts were identified as promising for bonding nylon to Gore-Tex. However, hydrostatic testing on sewn and sealed seams indicated that Michem 4990 had the most immediate promise. Therefore, the Michem resin was used for larger scale thread coating and evaluation.

* DuPont manufacturing process using looped yarn, two different yarn tensions.

SUMMARY

Commercial material suppliers were contacted for their recommendations on specific grades of hot melt resins (preferably low melting) for bonding nylon and/or polyester to Gore-Tex.[®] Sample laminates were prepared consisting of the substrate (nylon, polyester, Gore-Tex) and the adhesive. The peel strength of the adhesive versus substrate was evaluated dry and, if promising, immersed in water for 48 hours. Thread was coated on a laboratory scale with the most promising adhesives. Hand sewn samples were heated in an oven to melt the adhesive and screened for hydrostatic resistance with a laboratory pressure filter. The five most promising of these were evaluated for hydrostatic resistance as per Federal Standard 191A, TM 5512.

The adhesive chosen for pilot plant coating and machine sewing was the Michem 4990 based on the results of the hydrostatic resistance test.

A pilot plant scale coating apparatus was constructed to coat large amounts of thread for machine sewing. Fluorinert (Fluorocarbon - 3M) was used as a lubricant to aid in the sewing process. The sewability (small sewing machine) of the triple-coated Michem 4990 thread with the use of the lubricant was demonstrated and found to be very comparable to uncoated thread.

The results of the hydrostatic resistance testing were lower for samples sewn and sealed with the pilot plant-coated green thread than for those using the coated white thread, 12 to 14 psig versus an average 57 psig. The green thread was coated as received while the white thread was solvent washed prior to coating. These results suggest that a solvent wash may be required before coating the thread in order to obtain satisfactory hydrostatic resistance in the final sewn and sealed seams.

The following conclusions can be drawn from this Phase I program:

- * It is feasible to coat nylon thread with a hot melt resin, sew a shell fabric with the resulting thread, heat the seam briefly to effect a seal, and thereby prevent hydrostatic leakage in the area of the sewn and sealed seam.
- * Of the hot melt resins, Michem 4990, a water dispersion of ethylene-acrylic acid copolymer, provided sewn and sealed fabric laminates with the highest and most reproducible hydrostatic resistance.
- * Selected polyamide, urethane, ethylene-vinyl acetate and ethylene-acid hot melt resins also appear promising as nylon thread coatings for this application.
- * Sewability was demonstrated using nylon thread coated with the preferred resin and a small portable sewing machine.

PREFACE

The effort documented in this report was conducted by the following Springborn Laboratories (S/L) personnel:

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The author wishes to acknowledge the many helpful contributions of Mr. Steve Szczesuil, Project Officer, Natick Research, Development and Engineering Center.

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DEVELOPMENT OF HEAT-SEALABLE SEWING THREAD

I. INTRODUCTION

Developmental CP fabrics contain a waterproof breathable membrane (Gore-Tex)[®] barrier that is laminated to the underside of an outer shell fabric.¹ This barrier provides liquid water resistance and some degree of agent resistance. During sewing this barrier becomes perforated, inviting penetration by liquid and vaporous agents, thereby posing a severe hazard to the soldier. This problem is presently handled by using a hot melt tape to seal both the thread bundles and holes generated by sewing.

The purpose of this program was to demonstrate the feasibility of using a hot melt thread coating to seal nylon and polyester thread to a sewn Gore-Tex/outer shell fabric laminate. It was an objective that this hot melt coating, when reheated, would fill the needle holes and thread interstices so as to provide a barrier to vapors and liquids in sewn and sealed seam areas. Specifically, the sewn and sealed seams should provide a hydrostatic resistance of at least 35 psig, a criterion established by Natick RD&E under previous contract DAAK60-84-C-0052.²

This program addressed two basic areas of feasibility: (1) Is it possible to satisfactorily machine sew Gore-Tex/shell fabric laminate using a thread which has been coated with a hot melt resin? Will problems of softening resin or insufficient lubricity prevent realistic sewing speeds? and (2) Is it possible to heat the sewn seams and fuse the hot melt resin in such a way as to seal the thread bundles and surrounding needle holes? Can this be done at a temperature which will not adversely affect the cloth and thread and which will provide adequate hydrostatic resistance?

In pursuit of these objectives, a number of commercially available hot melt coatings were selected and evaluated for adhesion to nylon, polyester, and Gore-Tex, for their ability to be coated onto the thread, for surface lubricity and sewability, and for their ability to provide hydrostatic resistance when applied to a thread and used in a sewn and sealed seam. The most promising hot melt was used to coat pilot quantities of thread which were submitted to Natick RD&E for independent evaluation.

II. DEVELOPMENTAL

A. Materials

Fabric laminates, sewing thread and candidate hot melt coating resins were procured as follows:

Fabric Laminate: Natick RD&E Center provided a nylon Taslan^{*}/Gore-Tex/tricot knit fabric laminated as representative of the outer shell construction in a chemical protective garment.

Sewing Thread: Natick RD&E Center provided nylon thread, V-T-295 Type I, Class A, Size E, in both green and white and in various lengths for both laboratory investigation as well as initial pilot plant coating. For the coating of 2000 feet of thread, one spool of white nylon thread V-T-295, Type I, Class A (no sizing) was received from Coats and Clark.

Hot Melt Coatings: Candidate commercially available materials were selected and procured from vendors based on the following criteria:

- Likelihood, based on chemical structure, of providing good adhesion to nylon, polyester, and Gore-Tex.
- Good lubricity and low surface tack for sewability.
- Good water resistance.
- Low melt viscosity and good flow-out on heating for efficient sealing.
- Adaptable to production coating of thread. For reasons of environmental protection, organic solvent based coatings were avoided where possible, and water dispersion and direct hot melt coating systems were given preference.

Candidate hot melt coatings were selected from the following resin classes; polyester, polyamide, polybutylene, ethylene/acrylic or methacrylic acid copolymer, and vinylidene chloride based copolymers. A complete list of resin grades and manufacturers appears in the Appendix.

^{*} DuPont manufacturing process using looped yarn, two different yarn tensions.

B. Initial Screening of Hot Melt Coatings

1. Peel Strength Adhesion of the Hot Melt Coating to Nylon and Polyester

In order for the hot melt coatings to perform satisfactorily, they must have adequate adhesion to the sewing thread, either nylon or polyester. To develop quantitative data on peel strength adhesion, hot melt coatings were applied to nylon and polyester and tested for "T" peel. In order to develop basic adhesion data, flat films of nylon and polyester were substituted for the thread.

Film/hot melt/film test laminates were prepared as follows:

First, free-standing sheets of the hot melt coating candidates were prepared. Most resins were compression molded into 4- to 5-mil-thick sheets at 330°F using Teflon coated press plates to facilitate release. Where the coating had been supplied as a water dispersion, the dispersion was cast onto a glass plate and the air-dried film stripped from the glass.

These hot melt films were then used to prepare nylon-hot melt-nylon and polyester-hot melt-polyester peel test laminates. The nylon and polyester films were first solvent cleaned and the laminates were prepared again by compression molding at 350°F and 39 psig. These laminates were cut into 1/2-inch-wide strips for peel testing. For convenience in reporting, the grams/inch factor was utilized.

Peel strength was determined with the peel force measured at a 90-degree angle to the plane of the sample. Results were reported as the average force in grams per unit width of test laminate. These initial peel test results appear in Table 1. During peel testing, force values generally oscillate and the range of values obtained is presented.

The most promising laminates, based on the initial peel strength data, were immersed in water at room temperature for 48 hours. The samples were then removed and, while still wet, retested for peel strength.

Based on previous experience, a peel strength of less than 200 grams/inch of width was considered inadequate for the application. Such a low adhesive strength would likely result in delamination of the hot melt coating from the sewing thread in the end-item with possible loss of hydrostatic resistance in the sewn and sealed garment.

2. Peel Strength Adhesion of the Hot Melt Coating to Gore-Tex

In order for the hot melt coatings to perform satisfactorily they must also have adequate adhesion to the Gore-Tex barrier. Again, in order to develop quantitative data on peel strength adhesion, hot melt coatings were applied to Gore-Tex films and tested for "T" peel.

TABLE 1

Preliminary Candidate Screening - Peel Strength (1/2" Width Sample)

| Sample I.D. Number | Adhesive | Laminate ^a Material | Adhesive Failure ^f (g/in.) Range | Water Immersed at RT for 48 hrs (g/in.) |
|-----------------------|---|-----------------------------------|---|---|
| 22006-1 A | Vitel 10,202 | Nylon | 0 | h. |
| 22006-1 B | (polyester) | Nylon ^b | 0 | h. |
| 22006-1 C | | Mylar | 0 | h. |
| 22006-2 A | Vitel 4709A | Nylon | 0 | h. |
| 22006-2 B | (polyester) | Nylon ^b | 0 | h. |
| 22006-2 C | | Mylar | 2200-2400 | 300-1000 |
| 22006-3 A | Estane 5701 | Nylon | 100-230 | h. |
| 22006-3 C | (urethane) | Mylar | 10-20 | h. |
| 22006-4 A | Estane 5715 | Nylon | 100-400 | 140-260 |
| 22006-4 C | (urethane) | Mylar | 500-800 | 500-740 |
| 22006-5 A | Dow EAA 459 | Nylon | 250 | h. |
| 22006-5 C | (ethylene-acid) | Mylar | 0 | h. |
| 22006-6 A | Surlyn 9970 | Nylon | 100-140 | h. |
| 22006-6 C | (ionomer) | Mylar | 0 | h. |
| 22006-7 A | TPX 11-808 ^j | Nylon | 100-300 | h. |
| 22006-7 B | (polyamide) | Nylon | 80-300 | h. |
| 22006-7 C | | Mylar | 0 | h. |
| 22002-A-3 | Elvamide 8023 (polyamide) | Nylon ^b | 120-500 | h. |
| 22003-A | Elvamide 8061 ^j (polyamide) | Nylon ^b Mylar | 60-600 0 | 100-440-660 (Nylon tore) |
| 22004-3 | Elvamide 8063 (polyamide) | Nylon ^b | 100-180 | h. |
| 22005-A-1 | Bostik 7376 (polyester) | Nylon ^b Mylar | 40-60 0-60 | h. h. |

a. Molded and press-laminated 330^oF

b. Aged less than 7 days

c. Molding and laminating at 275^oFd. Molding and laminating at 300^oF

e. Brittle when wet

f. Number of experiments as shown in table

g. Peel values generally oscillate during test

h. Test eliminated due to substandard dry peel

i. Grams peel/inch width

j. Fluid melt

k. Yields unusually fluid melt

l. Received late in program

m. Poor friction characteristics, not tested further

TABLE 1 - Continued

Preliminary Candidate Screening - Peel Strength (1/2" Width Sample)

| Sample I.D. Number | Adhesive | Laminate ^a Material | Adhesive Failure ^f in grams/inch ^g Range | Water Immersed at RT for 48 hrs (gms/in.) |
|-----------------------|--|-----------------------------------|--|---|
| 22007-1 | Michem 4990 Prime (ethylene-acid dispersion) | Nylon | 100-850 | 300-360 |
| | | Mylar | 0 | h. |
| 22007-2 | Neo-Rez R960 | Nylon | 400-700 | 0-100 milky white |
| | | Mylar | 0 | appearance |
| 22007-3 | Duraflex DP 8910 (polybutylene) | Nylon ^c | 0 | h. |
| | | Nylon ^d | 0 | h. |
| | | Mylar ^c | 0 | h. |
| | | Mylar ^d | 0 | h. |
| 22008-1 | Nucrel 699 | Nylon | 18-240 | h. |
| | | Nylon | 300-420 | h. |
| | | Mylar | 0 | h. |
| | | Mylar | 0 | h. |
| 22008-2 | Nucrel 925 | Nylon | 300-550 | 400-420 |
| | | Nylon | 300-840 | h. |
| | | Mylar | 0 | h. |
| | | Mylar | 0 | h. |
| 22008-3 | Hytrel 4056 | Nylon | 100-150 | 60-160 |
| | | Nylon | 120-150 | 0 |
| | | Mylar | 300-460 | 1000-2000 |
| | | Mylar | 300-700 | 500-800 |
| 22008-4 | Bostick 7155 | Nylon | 20-30 | h. |
| | | Nylon | 20-30 | h. |
| | | Mylar | low (~20) | h. |
| | | Mylar | low (~20) | h. |

a. Molded and press-laminated 330°F

b. Aged less than 7 days

c. Molding and laminating at 275°F

d. Molding and laminating at 300°F

e. Brittle when wet

f. Number of experiments as shown in table

g. Peel values generally oscillate
during test

h. Test eliminated due to substandard dry peel

i. Grams peel/inch width

j. Fluid melt

k. Yields unusually fluid melt

l. Received late in program

m. Poor friction characteristics, not tested
further

TABLE 1 - Continued

Preliminary Candidate Screening - Peel Strength (1/2" Width Sample)

| Sample I.D. Number | Adhesive | Laminate ^a Material | Adhesive Failure ^f (g/in.) Range ^g | Water Immersed at RT for 48 hrs (g/in.) |
|-----------------------|-----------------------------|-----------------------------------|--|---|
| 22008-5 | Bostick 7178 | Nylon | 0 | h. |
| | | Nylon | 0 | h. |
| | | Mylar | 0 | h. |
| | | Mylar | 0 | h. |
| 22008-6 | Serfene 181 | Nylon | 500-550 | h. |
| | | Mylar | 0 | h. |
| 22008-7 | Serfene 2060 ^e | Nylon | 1000 | 300-400 ^e |
| | | Nylon | 600-800 (Nylon tore) | (Nylon tore) |
| | | Mylar | (Mylar tore) | >1000 |
| | | Mylar | 1000 | 0-100 100-220 |
| 22008-8 | Elvax 4310 | Nylon | 800-1900 | m. |
| | | Nylon | 1600-1900 | m. |
| | | Mylar | 220-400 | m. |
| | | Mylar | 240-400 | m. |
| 22009-1 | Macromelt 6301 ^j | Nylon | 400-1200 | l. |
| | | Nylon | 800-1300 | l. |
| | | Mylar | 0 | l. |
| | | Mylar | 0 | l. |
| 22009-2 | Macromelt 6071 ^j | Nylon | 500-800 | l. |
| | | Nylon | 400-800 | l. |
| | | Mylar | 400-500 | l. |
| | | Mylar | 300-700 | l. |
| 22009-3 | Macromelt 6030 | Nylon | 0 | h. |
| | | Nylon | 0 | h. |
| | | Mylar | 0 | h. |
| | | Mylar | 0 | h. |
| 22009-4 | Uni-Rez 2655 | Nylon | 800-1600 | 60-450 |
| | | Nylon | 800-1300 | 20-360 |

a. Molded and press-laminated 330°F

b. Aged less than 7 days

c. Molding and laminating at 275°F

d. Molding and laminating at 300°F

e. Brittle when wet

f. Number of experiments as shown in table

g. Peel values generally oscillate during test

h. Test eliminated due to substandard dry peel

i. Grams peel/inch width

j. Fluid melt

k. Yields unusually fluid melt

l. Received late in program

m. Poor friction characteristics, not tested further

Test laminates were prepared as follows:

First, free-standing sheets of the hot melt coating candidates were prepared as discussed on page 3, using either compression molding or casting of water dispersions.

These hot melt films were then used to prepare nylon/hot melt/Gore-Tex/hot melt/nylon and polyester/hot melt/Gore-Tex/hot melt/polyester peel test laminates. These laminates were prepared at 350 degrees F using a roll with a 10-pound weight. Higher pressures were avoided because they result in adhesive penetration of the Gore-Tex porosity, thereby yielding falsely high peel strength results. Samples were again cut into 1/2-inch-wide strips for peel testing.

Peel strength was determined with the peel force measured at a 90-degree angle to the plane of the sample. Results were reported as the average force in grams per unit width of test laminate. Samples were tested dry and after immersion in water at room temperature for 48 hours. The results appear in Table 2.

Following the wet Gore-Tex versus adhesive peel strength testing, it was sometimes possible to initiate separation between the cover film and the adhesive. This peel strength is also reported in Table 2, last column. Where separations could not be initiated, peel strength is tabulated as "high". This wet peel data may be more significant than the wet peel data of Table 1 since the Table 2 lamination was effected at a lower pressure, more representative of what would be encountered in the end-use.

C. Coated Thread - Laboratory Scale

1. Adhesive Formulations

The remainder of the development work was devoted to application of promising coatings to nylon thread and evaluation of the effects of the coatings on sewability and hydrostatic resistance of sewn and sealed seams. With the exception of Michem 4990, which is a water dispersion, the coating resins were formulated with appropriate solvents as well as, in some cases, a chemical blowing agent. The blowing agent created a closed cell foam which when expanded would aid in sealing the holes with minimal quantities of adhesive melt. The formulations appear in Table 3.

2. Initial Thread Coating

For the initial work, soft finish nylon thread³ was Soxhlet extracted for 24 hours to remove any silicone oil lubricants, or other agents, used to treat the thread.

TABLE 2

Gore-Tex Laminations - Peel Strength (1/2" Width Samples)

| Samples I.D. Number | Laminate System | Gore-Tex vs Adhesive Dry ^{a.} f. (g/in.) | Gore-Tex vs Adhesive Dry ^{c.} (g/in.) | Gore-Tex vs Adhesive ^{c.} Wet ^{c.} (g/in.) | Adhesives vs Cover Film Only ^{a.} Wet ^{a.} (g/in.) |
|------------------------|--|--|--|--|--|
| 22010-1 | Nylon-8061-Gore-Tex-8061-Nylon | 420-440 ^{d.} 380-520 | 380 | 190-400 | 240-400 180-300 |
| 22010-2 | Mylar-87061-Gore-Tex-8061 Mylar | 500-620 440-980 | 500 | 300-340 | 0 0 |
| 22010-3 | Nylon-Hytrel 4056-Gore-Tex- Hytrel 4056-Nylon | 160-300 350-370 | 360 | 180-220 | 20-40 40-80 |
| 22010-4 | Nylon-Nucrel 925-Gore-Tex- Nucrel 925-Nylon | 260-520 280-500 | 420 | 240-430 | 140 170-200 |
| 22010-5 | Nylon-Michem 4990-Gore-Tex- Michem 4990-Nylon | 330-440 420-460 | 460 | 240-460 | > 420 > 460 |
| 22010-6 | Nylon-Estane 5715-Gore-Tex- Estane 5715-Nylon | 220-400 420-520 | 450 | 220-260 | > 400 > 520 |
| 22010-7 | Nylon-R960-Gore-Tex- R960-Nylon | - 0 - 0 | e. e. | e. e. | - 0 - 0 |
| 22010-8 | Mylar-Estane 5715-Gore-Tex- Estane 5715-Mylar | 240-300 220-240 | 300 | 220-360 | 100 - 0 |
| 22010-9 | Mylar-Vitel 4709A-Gore-Tex- Vitel 4709A-Mylar | 470 640-800 | 460 | 420 | high ^{b.} high |
| 22010-10 | Mylar-Hytrel 4056-Gore-Tex- Hytrel 4056-Mylar | 0-20 0-60 | e. e. | e. e. | 200-400 300-700 |
| 22010-11 | Mylar-Serfene 2060-Gore-Tex- Serfene 2060-Mylar | 0 0 | e. e. | e. e. | 40-60 60-100 |
| 22010-12 | Nylon-Serfene 2060-Gore-Tex- Serfene 2060-Nylon | 0-100 60-120 | e. e. | e. e. | 120-220 140-220 |
| 22010-13 | Nylon-TPX 11-808-Gore-Tex- TPX11-808-Nylon | 770-800 400-480 | 480 | 330-370 | high ^{b.} high |
| 22010-14 | Nylon-Elvax 4310-Gore-Tex- Elvax 4310-Nylon | 300-380 300-330 | 400 | 320-300 | high ^{b.} high |
| 22010-15 | Nylon-Macromelt 6071-Gore-Tex- Macromelt 6071-Nylon | 220-260 140-260 | 120 | e. | high ^{b.} high |
| 22010-16 | Nylon-Macromelt 6301-Gore-Tex- Macromelt 6301-Nylon | 300-360 290-380 | 330-400 | 340-360 | high ^{b.} high |

a. Sample strips #1 and #2 (duplicate)

b. Cannot initiate failure between cover
film and adhesive

c. Sample strip #3

d. Peel values generally oscillate during test

e. Test eliminated due to substandard dry peel

f. Grams peel/inch width

TABLE 3

Thread Coating Formulations

| Sample I.D. Number | Adhesive | Formulation |
|-----------------------|---|--|
| 22014-1 ^a | (Not adhesive) Celogen OT (Blowing Agent) | 48.8 gms Celogen OT, 0.18 gms Tamol 850, 37.3 gms H ₂ O D.I., 0.10 gms R&H CF-10 (ball mill for 24 hrs) |
| 22014-2 | TPX 11-808 | 7 gms TPX 11-808, 11 gms IPA (isopropyl alcohol), 10 gms Toluene (dissolve at 80°C - capped) |
| 22014-4 | Elvamide 8061 | 7 gms Elvamide 8061, 10.5 gms methanol, 10.5 gms methylene chloride (dissolve 40°C - capped) |
| 22016-1 | TPX 11-808 & Blowing Agent | 7 gms TPX 11-808, 0.14 gms Celogen OT, 11 gms IPA, 0.07 gms Aerosol OT 100%, 10 gms Toluene |
| 22016-2 | Elvamide 8061 & Blowing Agent | 7 gms, Elvamide 8061, 0.14 gms Celogen OT, 10.5 gms methanol, 0.07 gms Aerosol OT 100%, 10.5 gms methylene chloride |
| 22016-3 | Michem 4990 & Blowing Agent | 28.0 gms Michem 4990, 0.565 gms #22014-1, 0.098 gms Aerosol OT 100% |
| 22016-4 | Estane 5715 | 20 gms Estane 5715, 80 gms MEK (methyl ethyl ketone) |
| 22016-5 | Estane 5715 & Blowing Agent | 20 gms Estane 5715, 0.4 gms Celogen OT, 80 gms MEK, 0.2 gms Aerosol OT 100% |
| 22016-6 | Macromelt 6071 | 28 gms Macromelt 6071, 44 gms IPA, 41 gms Toluene |
| 22016-7 | Macromelt 6301 | 28 gms Macromelt 6301, 44 gms IPA, 40 gms Toluene |
| 22021-1 | Macromelt 6301 & Blowing Agent | 56.0 gms formulation #22016-7, 0.28 gms Celogen OT, 0.14 gms Aerosol OT 100% |
| 22021-2 | Macromelt 6071 & Blowing Agent | 56.0 gms formulation #22016-6, 0.28 gms Celogen OT, 0.14 gms Aerosol OT 100% |

a. This formulation is a water dispersion of the blowing agent for
use in #22016-3

Three-foot lengths of thread were coated on a laboratory scale for initial screening. The thread was pulled through an adhesive solution and up through a 22-mil die to remove excess adhesive. The thread was initially dried at room temperature for 20 minutes and oven dried at 110°C for 20 minutes. The thread was then weighed for coating add-on. To achieve a heavier add-on, the dry thread was recoated a second or third time. Because the coating die diameter was constant, as the thread width increased the coating weight per application generally decreased.

3. Initial Hand Sewing

Adhesives evaluated for initial hand sewing were selected (Table 3) from the results (Tables 1 and 2) of peel strength versus substrates. Candidate resins were not only chosen for their adhesive properties, but also for their ability to flow out at 350°F. Promising resins were allowed to melt on a 350°F-/177°C hot plate and resins with good flow out/low melt viscosity were selected. The resins were also screened with the use of a blowing agent in the formulation. The blowing agent creates a foam which helps seal the holes with minimal quantities of adhesive melt (Table 3).

Two-inch-diameter circles were cut from nylon Taslan/Gore-Tex/tricot knit cloth laminate material⁴ and were sewn with the coated thread. Initial sewing was done with a single thread using a hand sewing needle, three stitches per circle, as illustrated by Figure 1. Subsequent hand sewing included: double thread i.e. Figure 2 and quadruple thread, i.e., Figure 3. Some hand stitching was undertaken with a sewing machine needle, leaving a double thread in the hole, i.e., Figure 4. The purpose of two and four threads passing through the needle hole is to simulate a sewing machine stitch wherein two to four threads will pass through the hole (see Figure 5, page 13). Note that six needle holes see the hydraulic pressure, and leakage will occur through the least well-sealed area.

The sewn circles were then placed in a 350°C oven for 30 minutes to allow the hot melt adhesive to melt, wick out, and seal the holes. The formulations containing a blowing agent were placed in the oven for 12 minutes. Previous studies at Springborn Laboratories⁵ have shown that a longer time causes the foam that forms to collapse after approximately 12 minutes.

The samples were then subjected to water under pressure using a laboratory pressure filter. The samples were mounted in the pressure filter bracket and the device filled with water and subjected to pressure. Pressure was applied at a rate of 1 psi/second. The pressure is recorded when water begins to drip from the funnel exit. This device was only used for initial screening. Results are presented in Tables 4, 5, 6, 7 and summarized in Table 8.



Cross Section of Single
Thread in Needle Hole

Figure 1: Test Specimen, Three Stitch,
Single Thread



Cross Section of Double
Thread in Needle Hole

Figure 2: Test Specimen, Three Stitch,
Double Thread



Cross Section of Quad
Thread in Needle Hole

Figure 3: Test Specimen, Three Stitch,
Quadruple Thread

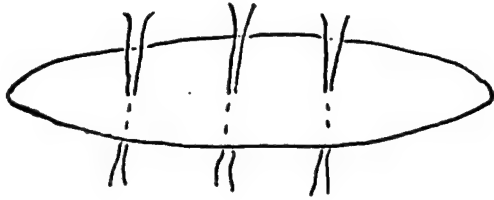
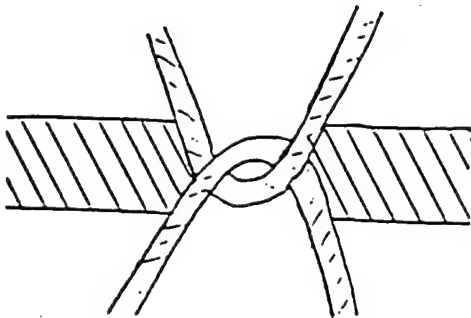


Figure 4: Test Specimen, Sewing Machine Needle,
Three Stitch, Double Thread



Cross Section of Sewing
Machine Stitch

Figure 5: Sewing Machine Stitch, Loop Within
Needle Hole (Type 301)

TABLE 4

Preliminary Screening - Pressure Filter Test
(Single Thread)^b

| Sample I.D. Number | Adhesive | Wt. of 1' Thread (gms) | Wt. Coating & Thread (gms) | % Weight Add-on | # of Dips | Pressure ^a Test (psi) |
|-----------------------|----------------------------|---------------------------|-------------------------------|--------------------|--------------|-------------------------------------|
| Control | None | 0.0235 | - | - | - | Gravity Leak < 1 |
| 22015-1A | TPX 11-808 | 0.0235 | 0.0248 | 5.2 | 1 | 10 |
| 22015-1B | 25% Solids | 0.0235 | 0.0257 | 8.5 | 2 | 5 |
| 22015-1C | Formulation. # 22014-2 | 0.0235 | 0.0274 | 14.2 | 3 | 9 |
| 22017-4A | TPX 11-808 | 0.0235 | 0.0281 | 16.37 | 1 | 12 |
| 22017-4B | W/Blowing | 0.0235 | 0.0319 | 26.33 | 2 | 14 |
| 22017-4C | Agent Formula. #22016-1 | 0.0235 | 0.0351 | 33.05 | 3 | 16 |
| 22015-3A | 8061 | 0.0235 | 0.0310 | 24.2 | 1 | 10 |
| 22015-3B | Formulation | 0.0235 | 0.0368 | 36.14 | 2 | 10 |
| 22015-3C | #22014-4 | 0.0235 | 0.0400 | 41.25 | 3 | 10 |
| 22017-5A | 8061 w/Blowing | 0.0235 | 0.0300 | 21.66 | 1 | 14 |
| 22017-5B | Agent Formula. | 0.0235 | 0.0358 | 34.35 | 2 | 10 |
| 22017-5C | #22016-2 | 0.0235 | 0.0429 | 42.22 | 3 | 10 |
| 22017-6A | Macromelt 6071 | 0.0235 | 0.0292 | 19.52 | 1 | 11 |
| 22017-6B | Formulation | 0.0196 | 0.0279 | 28.42 | 2 | 13 |
| 22017-6C | #22016-6 | 0.0235 | 0.0379 | 37.99 | 3 | 13 |
| 22022-2A | Macromelt 6071 | 0.0235 | 0.0295 | 20.33 | 1 | 13 |
| 22022-2B | w/Blowing | 0.0235 | 0.0376 | 37.5 | 2 | 13 |
| 22022-2C | Agent Formula. #22021-2 | 0.0235 | 0.0388 | 39.43 | 3 | 15 |
| 22017-7A | Macromelt 6301 | 0.0235 | 0.0292 | 19.52 | 1 | 11 |
| 22017-7B | Formulation | 0.0235 | 0.0337 | 30.26 | 2 | 12 |
| 22017-7C | #22016-7 | 0.0235 | 0.0376 | 37.5 | 3 | 12 |
| 22015-5A | Michem Prime | 0.0235 | 0.0333 | 29.4 | 1 | 8 |
| 22015-5B | 4990 Neat | 0.0235 | 0.0355 | 33.8 | 2 | 8 |
| 22015-5C | | 0.0235 | 0.0400 | 41.25 | 3 | 10 |
| 22017-3A | Michem Prime | 0.0235 | 0.0300 | 21.66 | 1 | 10.5 |
| 22017-3B | 4990 w/Blowing | 0.0235 | 0.0331 | 29.03 | 2 | 10 |
| 22017-3C | Agent Formula. #22016-3 | 0.0235 | 0.0391 | 39.89 | 3 | 12 |
| 22017-1A | Estane 5715 | 0.0235 | 0.0280 | 16.07 | 1 | 0 |
| 22017-1B | Formulation | 0.0235 | 0.0329 | 28.57 | 2 | 7.5 |
| 22017-1C | #22016-4 | 0.0235 | 0.0371 | 36.65 | 3 | 8 |
| 22017-2A | Estane 5715 | 0.0235 | 0.0279 | 15.77 | 1 | 11 |
| 22017-2B | w/Blowing Agent | 0.0235 | 0.0333 | 29.42 | 2 | 10 |
| 22017-2C | Formu.#22016-5 | 0.0235 | 0.0366 | 35.79 | 3 | 10 |

a. Six threaded needle holes per test; number of experiments as shown in table

b. As in Figure 1

TABLE 5

Pressure Filter Test - Double Thread^c.

| Sample I.D. Number | Adhesive | Wt. of 1' Thread (gms) | Wt of Coating & Thread (gms) | Percent Wt. Add-on | # of Dips | Pressure Test a. (psig) ^b . |
|--------------------|---|------------------------|------------------------------|--------------------|-----------|--|
| 22024-1 | TPX 11-808 Formulation #22014-2, Thread #22015-1C | 0.0235 | 0.0274 | 14.2 | 3 | 14 |
| 22022-3 | TPX 11-808 w/ Blowing Agent Formulation #22016-1, Thread # 22017-4C | 0.0235 | 0.0351 | 33.05 | 3 | 18 |
| 22022-4 | Macromelt 6071 Formulation #22016-6, Thread #22017-6C | 0.0235 | 0.0379 | 37.99 | 3 | 14 |
| 22022-6 | Macromelt 6071 w/Blowing Agent, Formulation #22021-2, Thread #22022-2C | 0.0235 | 0.0388 | 39.43 | 3 | 27 |
| 22022-5 | Macromelt 6301 Formulation #22016-7, Thread #22017-7C | 0.0235 | 0.0376 | 37.5 | 3 | 23 |
| 22024-3 | Michem Prime 4990 Neat, Thread #22015-5C | 0.0235 | 0.0400 | 41.25 | 3 | 13 |
| 22024-5 | Michem Prime 4990 w/ Blowing Agent Formulation #22016-3, Thread #22017-3C | 0.0235 | 0.0391 | 39.89 | 3 | 18 |
| 22024-4 | Estane 5715 w/Blowing Agent Formulation #22016-5, Thread #22017-2C | 0.0235 | 0.0366 | 35.79 | 3 | 14 |
| 22024-6 | 8061 w/ Blowing Agent Formulation #22016-2, Thread #32017-5C | 0.0235 | 0.0429 | 42.22 | 3 | 12 |

a. Heat 30 minutes 117°C before testing;
12 minutes heating for foam formulations

b. Six threaded needle holes per test; number of experiments as shown in table

c. As in Figure 2

TABLE 6

Pressure Filter Test - Double Thread
(Sewing Machine Needle Puncture) ^a.

| Sample I.D. Number | Adhesive | Wt. of 1' Thread (gms) | Wt. of Coating & Thread (gms) | Percent Wt. Add-on | Number of Dips | Pressure Test (psi) ^c . |
|-----------------------|---|------------------------------|-------------------------------------|-----------------------|-------------------|--|
| 22025-1A | Macromelt 6301 Formulation #22016-7 | 0.0235 | 0.0387 | 39.2 | 3 | 14 |
| 22025-2A | Macromelt 6071 w/ Blowing Agent , Formulation #22021-1 | 0.0235 | 0.0364 | 35.4 | 3 | 14 ^b . |
| 22025-3A | Macromelt 6071 Formulation #22016-6 | 0.0235 | 0.0375 | 37.3 | 3 | 15 |
| 22025-4A | TPX 11-808 w/ Blowing Agent Formulation #22016-1 | 0.0235 | 0.0375 | 37.3 | 3 | 17 ^b . |
| 22025-5A | Michem Prime 4990 (neat) | 0.0235 | 0.0432 | 45.6 | 3 | 25 |
| 22025-6A | Michem Prime 4990 w/ Blowing Agent Formulation #22016-3 | 0.0235 | 0.0412 | 42.9 | 3 | > 40 |

a. Hand sewn as in Figure 4

b. Possible foam collapse (in oven over 12 minutes)

c. Six threaded needle holes per test; number of experiments as shown in table

TABLE 7

Pressure Filter Test - Quadruple Thread^c

| Sample I.D. Number | Adhesive | Wt. of 1' Thread (gms) | Wt. of Coating & Thread (gms) | % Weight Add-on | Number of Dips | Pressure Test b. (psi) |
|-----------------------|--|------------------------------|-------------------------------------|--------------------|-------------------|------------------------------|
| 22025-1B | Macromelt 6301 Formulation #22016-7 | 0.0235 | 0.0380 | 38.15 | 3 | 14 |
| 22025-2B | Macromelt 6071 w/Blowing Agent Formulation #22021-1 | 0.0235 | 0.0368 | 36.14 | 3 | 8 ^a |
| 22025-3B | Macromelt 6071 Formulation #22016-6 | 0.0235 | 0.0374 | 37.1 | 3 | 32 |
| 22025-4B | TPX 11-808 w/Blowing Agent Formulation #22016-1 | 0.0235 | 0.0375 | 37.3 | 3 | 4 ^a |
| 22025-5B | Michem Prime 4990 (Neat) | 0.0235 | 0.0435 | 45.9 | 3 | > 45 |
| 22025-6B | Michem Prime 4990 w/Blowing Agent Formulation #22016-3 | 0.0235 | 0.0423 | 44.4 | 3 | 38 |

a. Possible Foam Collapse
(in oven over 12 minutes)

b. Six threaded needle holes per test;
number of experiments as shown in table

c. As in Figure 3

TABLE 8

Summary, Pressure Filter Tests^{c.}

| Adhesive | Sample I.D. Number | Hydrostatic Pressure Resistance (psi) ^{b.} | | | | % Weight Add-on per Thread |
|--|-----------------------|---|--------------------------------|---|-----------------------------------|----------------------------------|
| | | Single ^{d.} Thread | Double ^{e.} Thread | Double ^{f.} Thread Machine Needle | Quadruple ^{g.} Thread | |
| TPX 11-808 w/ Blowing Agent, Formulation #22016-3 | 22017-4C | 16 | - | - | - | 33.05 |
| | 22022-3 | - | 18 | - | - | 33.05 |
| | 22025-4A | - | - | 17 ^{a.} | - | 37.3 |
| | 22025-4B | - | - | - | 4 ^{a.} | 37.3 |
| Macromelt 6301 Formulation #22016-7 | 22017-7C | 12 | - | - | - | 37.5 |
| | 22022-5 | - | 23 | - | - | 37.5 |
| | 22025-1A | - | - | 14 | - | 39.2 |
| | 22025-1B | - | - | - | 14 | 38.15 |
| Macromelt 6071 w/Blowing Agent Formulation #22021-2 | 22022-2C | 15 | - | - | - | 39.43 |
| | 22022-6 | - | 27 | - | - | 39.43 |
| | 22025-2A | - | - | 14 ^{a.} | - | 35.4 |
| | 22025-2B | - | - | - | 8 ^{a.} | 36.14 |
| Macromelt 6071 Formulation #22016-6 | 22017-6C | 13 | - | - | - | 19.52 |
| | 22022-4 | - | 14 | - | - | 37.99 |
| | 22025-3A | - | - | 15 | - | 37.3 |
| | 22025-3B | - | - | - | 32 | 37.1 |
| Michem Prime 4990 (Neat) | 22015-5C | 10 | - | - | - | 41.25 |
| | 22024-3 | - | 13 | - | - | 41.25 |
| | 22025-5A | - | - | 25 | - | 46.6 |
| | 22025-5B | - | - | - | > 45 | 45.9 |
| Michem Prime 4990 w/Blowing Agent Formulation #22016-3 | 22017-3C | 12 | - | - | - | 39.89 |
| | 22024-5 | - | 18 | - | - | 39.89 |
| | 22025-6A | - | - | > 45 | - | 42.9 |
| | 22025-6B | - | - | - | 38 | 44.4 |

a. Possible foam collapse
(in oven over 12 minutes)

b. Six threaded needle holes per
test; number of experiments
as shown in table

c. Data taken from Tables 4,5,6 and 7

d. As in Figure 1

e. As in Figure 2

f. As in Figure 4

g. As in Figure 3

D. Evaluation of Volatile Yarn Lubricants

Friction between the coating and the metal needle may cause heating of the needle resulting in a softening of the coating. This in turn would impede the sewing process. It was thought that the friction caused by the coating could be reduced by the use of a volatile lubricant.

Preliminary friction measurements were carried out using an 860-g steel bar attached to a spring scale. The film to be screened was placed on a glass strip with rounded side edges, and the metal weight was pulled along its surface. Friction was measured in grams of force required to move the steel bar across the film. The results appear in Table 9.

Lubricants were evaluated in a friction test using films of Michem 4990[®] Prime. Table 10 shows the comparison of the dry film with a lubricated film.

E. Hydrostatic Pressure Resistance

Five of the most promising candidate formulations were coated onto thread and evaluated^{*} for hydrostatic resistance per Test Method 5512.⁶ Candidate resins were chosen for their adhesion to nylon and/or Mylar and Gore-Tex, as well as the results of the initial pressure filter.

The thread was coated (3 dips) and hand-sewn into the cloth as double thread. The samples were then placed in a 350°F oven for 30 minutes except for the blowing agent formulations which only remained at 350°F for 12 minutes. Results are presented in Table 11.

*-----
* This test procedure was carried out by U.S. Testing Co., Inc.

TABLE 9

Friction Test - Adhesive vs Metal

| <u>Adhesive</u> | <u>Friction - Grams</u> ^{a.} |
|-----------------|---------------------------------------|
| Serfene 2060 | 160, 170, 170 |
| Vitel 4709-A | 170, 170, 180 |
| Elvamide 8061 | 180, 190, 180 |
| TPX 11-808 | 180, 180, 200 |
| Estane 5715 | 180, 200, 190 |
| R-960 | 280, 250, 260 |
| Nucrel 925 | 310, 300, 320 |
| Michem 4990 | 350, 370, 400 |
| Elvax 4310 | 550, 500, 550 |
| Hytrel 4056 | 600, 620, 650 |

a. Triplicate determinations

TABLE 10

Michem Prime 4990Friction Test w/Various Lubricants

| <u>Lubricant</u> | <u>Friction - Grams</u> | |
|--|-------------------------|-----------------------------------|
| | <u>Dry (Control)</u> | <u>w/ Lubricant</u> ^{c.} |
| 3M FC-70 (Fluorocarbon) | 350 | 50 - 150 |
| Union Carbide Volatile Silicone 7158 | 300 | 175 - 300 (200 Avg) |
| 2% Polyvinylpyrrolidone (PVP) ^{a.} K-90 GAF | 375 | 175 - 300 (200 Avg) |
| 2% PVP + 1% Union Carbide Silwet L-77 ^{a.} | 400 | 100 - 300 (200 Avg) |
| 2% PVP + 1% Air Products Surfynol 104 ^{a.b.} | 400 | 100 - 200 |
| 1.5% PVP + 0.25 Surfynol 104 ^{a.} | 400 | 200 - 300 (250 Avg) |

^{a.} Aqueous solution^{b.} Not entirely soluble^{c.} Duplicate determinations

TABLE 11
Hydrostatic Pressure Resistance

| Sample I.D. # | Adhesive (Formulation #) | % Wt. Add-on | Hydrostatic ^a Pressure Resistance (psi) | | |
|---------------|---|--------------|---|---------|--------|
| | | | Individual | Average | Median |
| 22029-1A | Control | - - | 74 | 93 | 98 |
| 22029-1B | (Unsewn circles) (Cloth as received) | | 100 | | |
| 22029-1C | | | 104 | | |
| 22029-1D | | | 88 | | |
| 22029-1E | | | 98 | | |
| 22029-2A | Uncoated | - - | 0 | 0 | 0 |
| 22029-2B | Thread | | 0 | | |
| 22029-2C | | | 0 | | |
| 22029-2D | | | 0 | | |
| 22029-2E | | | 0 | | |
| 22027-1A | Michem | 44.7 | 28 | 57 | 80 |
| 22027-1B | 4990 | | 7 | | |
| 22027-1C | Neat | | 84 | | |
| 22027-1D | #22026-1 | | 86 | | |
| 22927-1E | | | 80 | | |
| 22027-2A | Michem | 41.3 | 10 | 28 | 10 |
| 22027-2B | 4990 | | 30 | | |
| 22027-2C | w/blowing | | 10 | | |
| 22027-2D | agent | | 78 | | |
| 22927-2E | #22026-2 | | 10 | | |
| 22027-3A | Macromelt | 49.4 | 6 | 10 | 10 |
| 22027-3B | 6301 | | 10 | | |
| 22027-3C | #22026-3 | | 10 | | |
| 22027-3D | | | 16 | | |
| 22027-3E | | | 10 | | |
| 22027-4A | Macromelt | 53.6 | 0 | 16 | 8 |
| 22027-4B | 6301 | | 64 | | |
| 22027-4C | w/blowing | | 0 | | |
| 22027-4D | agent | | 8 | | |
| 22027-4E | #22026-8 | | 8 | | |
| 22027-5A | Uni-Rez | 37.9 | 0 | 4 | 6 |
| 22027-5B | 2655 | | 6 | | |
| 22027-5C | #22026-7 | | 0 | | |
| 22027-5D | | | 6 | | |
| 22027-5E | | | 6 | | |

a. Double thread, three stitches in each of five circles
Fed. Std. 191A, Method 5512

III. DISCUSSION OF RESULTS

A. Initial Screening

1. Peel Strength Adhesion of the Hot Melt Coatings to Nylon and Polyester

The peel strength results of the initial screening, Table 1, indicated that the hot melts with the greatest adhesion to nylon and polyester were as follows:

For Nylon

Estane 5701 [®]
Estane 5715 [®]
Dow EAA 459 [®]
TPX 11-808 [®]
Elvamide 8061 [®]
Michem 4990 [®]
Nucrel 699 [®]
Nucrel 925 [®]
Serfene 2060 [®]
Serfene 181 [®]
Elvax 4310 [®]
Macromelt 6301 [®]
Macromelt 6071 [®]
Unirez 2655 [®]
NeoRez R960 [®]

For Polyester

Vitel 4709A [®]
Estane 5715 [®]
Hytrel 4056 [®]
Serfene 2060 [®]
Elvax 4310 [®]
Macromelt 6071 [®]

To shorten the candidate list, those adhesives with relatively poor peel strength were dropped, for example, since the Nucrels are all chemically similar and Nucrel 925 provided better peel strength than Nucrel 699, the latter was dropped. Similarly, Serfene 2060 appeared to be superior to Serfene 181 and Estane 5715 was more promising than Estane 5701.

Hot melt laminates showing good peel strength were soaked 48 hours in water and retested, last column (Tables 1 and 2). Table 1 wet peel strength values, with few exceptions, were higher than Table 2 wet laminate values. This can be attributed to the higher lamination pressure used for the initial (Table 1) lamination.

Promising hot melts at this point included:

For Nylon

Estane 5715
Elvamide 8061
Michem 4990
Nucrel 925
Uni-Rez 2655
TPX 11-808
Elvax 4310
Macromelt 6301
Macromelt 6071

For Polyester

Estane 5715
Hytrel 4056
Vitel 4709A

2. Peel Strength Adhesion of the Hot Melt Coating to Gore-Tex

Peel strength data for Gore-Tex laminates, dry and wet are presented in Table 2. Additional data on the peel strength of laminates after water immersion is also included in Table 2, in last column. On the basis of these results, the more promising adhesives at this point were:

Nylon to Gore-Tex

Elvamide 8061
Nucrel 925
Michem 4990
Estane 5715
TPX 11-808
Elvax 4310
Macromelt 6071
Macromelt 6301

Polyester to Gore-Tex

Vitel 4709A

3. Evaluation of Coated Thread

To reduce the number of candidates, Nucrel and Elvax were dropped at this time on the basis of their poor friction characteristics (Table 9). Michem 4990 has relatively poor friction characteristics, but was retained as a candidate because of its very fluid melt characteristics.

The coated thread described in Section II-C of this report and in Table 3 was sewn in place with a hand stitching needle and heated to melt the coating. Sewn and sealed samples were subjected to hydrostatic testing.

Table 4 shows the results of samples sewn with a single thread at different coating weight add-ons. The control was a piece of cloth sewn with uncoated thread in the same manner as the samples using the coated thread. The control leaked water simply by gravity.

For most of the candidate resins, the results show an increase in pressure resistance with the use of a chemical blowing agent. The blowing agent was used to create a closed cell foam from the coating.

One would expect better sealing properties with increased coating weight, but this was not true for four of the experiments. For the single thread experiments, reported in Table 4, coating weight of over 20 to 28-percent resulted in little or no improvement in hydrostatic resistance.

Table 5 reports hydrostatic resistance results of the candidate coatings using a double thread, i.e. Figure 2. The results are substantially improved over those using a single thread.

It was thought that the hand-sewing needle technique was more damaging to the cloth than would be the case with a sewing machine needle. Therefore, stitches were made in the cloth with a sewing machine needle (size 16, 0.040-inch diameter) puncture. The thread was then drawn through this hole, Figure 4. The hydrostatic resistance results (Table 6) improved with some but not all of the samples over those using the hand sewing needle.

The samples were also hand-sewn using four threads passing through the hole to simulate a sewing machine stitch wherein the loop is within the needle hole (Figure 5) rather than above or below the needle hole. Table 7 presents pressure filter results with four threads per needle hole.

Table 8 shows a comparison of the results in Tables 4 to 7. Note that the "A" designation means 1 dip, "B" is 2 dips, and "C" or no letter is 3 dips of the coating. Using thread with Macromelt 6301 and Michem 4990, neat (no foaming agent), the quad (quadruple) thread provided the best hydrostatic resistance. One might expect that four threads would "fill" the needle hole better than two threads, i.e. see Figure 3 cross section versus Figure 4 cross section.

Although a double thread should be more difficult to seal against high pressure than a quadruple thread, Michem 4990 (22025-5A and 22025-6A, Table 8) showed good initial promise in sealing sewing machine needle puncture holes.

A dilute methyl violet/fluorocarbon detergent dye solution was used as a test vehicle to determine where the leaks had originated. Following the pressure filter test, the samples were dried and the stitches on the camouflage side of the sewn samples were painted with the dye solution. The reverse side of the sample was observed for penetration of the dye around the needle holes, or the dye penetrating the coating and wicking up via the inside of the thread bundle.

The TPX 11-808 samples showed penetration of the dye around the holes, but not discoloring the coated thread. The Elvamide 8061 did show discoloration through the braid of the thread, indicating penetration of the coating. The Michem 4990 and the Macromelt 6071 never showed penetration of any kind. The dye did not penetrate the intact nylon Taslan/Gore-Tex/tricot knit cloth laminate.

Lengths of thread coated with final candidate hot melts were sewn into barrier cloth, heat-fused and subjected to hydrostatic resistance per Test Method 5512, Table 11. Michem 4990 yielded the most consistent pressure resistance and the highest leak pressures. Hydrostatic results for coated thread using foaming agent were erratic, ranging from good to poor.

4. Pilot Plant Coated Thread/Machine Sewing

The Pilot plant-coated thread was coated as received with Michem 4990. No particular difficulty was encountered although, to obtain a well-fused coating, a significant amount of heat was needed in the drying section. The coated thread gave no handling problem when used on a small sewing machine operating at maximum speed (approximate 10 feet/minute).

Heat-fused machine-stitched barrier cloth, however, leaked at 12-14 psi (laboratory pressure filter). The solvent-extracted laboratory-coated white thread, on the other hand, when hand sewn and sealed gave stitches (Figure 5) which leaked at >35 psi. Thus, it would appear that special lots of thread without sizing will have to be obtained and utilized in the future for pilot plant coating.

5. Volatile Yarn Lubricants

The data presented in Table 9 indicates that some of the better hot melt adhesives (e.g. Michem 4990) have rather high friction characteristics against steel. This could result in softening/melting problems at very high sewing speeds, although the newer friction-resistant needles and the use of air cooled needles may prevent such problems. In the event friction problems arise, some precautionary work was undertaken to identify volatile lubricants that would enable high sewing speeds, but would evaporate before interfering with the bonding step.

The Table 10 results indicate that 3M's volatile fluorocarbon is by far the most efficient lubricant. Less expensive lubricants such as silicone, or dilute aqueous polyvinyl pyrrolidone, appear to be helpful but are inferior to the fluorocarbon.

B. Most Promising Candidate

Michelman Chemical's Michem Prime 4990 is an aqueous alkaline dispersion of Dow Chemical's Primacor Resin (EAA - ethylene acrylic acid). It was chosen for its performance in overall initial screening and from final hydrostatic resistance results.

1. Thread Coating

A pilot plant thread coating apparatus was assembled to coat the larger amounts of thread needed for machine sewing. A green nylon thread similar to the white soft finish thread was obtained in larger quantities and was used as received. (Solvent washing tended to extract much of the color). A drawing of the apparatus is presented in Figure 6. The thread was routed through the adhesive solution and pulled up through a 22-mil die to remove the excess adhesion. The thread was then passed through an "air-circulating oven" at -350°F to dry, followed by two fusing steps with infrared lamps, and the thread was then finally wound on a take-up spool. This process was repeated with previously coated thread to obtain higher weight add-ons.

There were initial problems with the coating on the thread being easily removed, (e.g., could be scraped off with a sharp object). Improvements in the drying and fusing process solved this problem with minimal difficulty. The oven temperature gradient was raised and the thread was moved closer to the IR lamps.

2. Machine Sewing

A standard 301 Lockstitch Singer sewing machine was used to test the coated thread for sewability. This is a commercially available household type machine, equipped with a size 18 (0.044-inch-diameter) needle.

Double-coated and triple-coated threads were evaluated as well as a control (uncoated thread) for sewability. The sewability was measured by recording sewing speed on a one-foot section of material. The uncoated thread stitched (full speed) at the rate of 11.5 feet/minute. The double-coated thread stitched almost as fast, 9.8 feet/minute. The triple-coated thread stitched at the rate of 9.7 feet/minute. All coated threads were sewn with the use of FC-70, a 3M volatile fluorocarbon, as a lubricant.

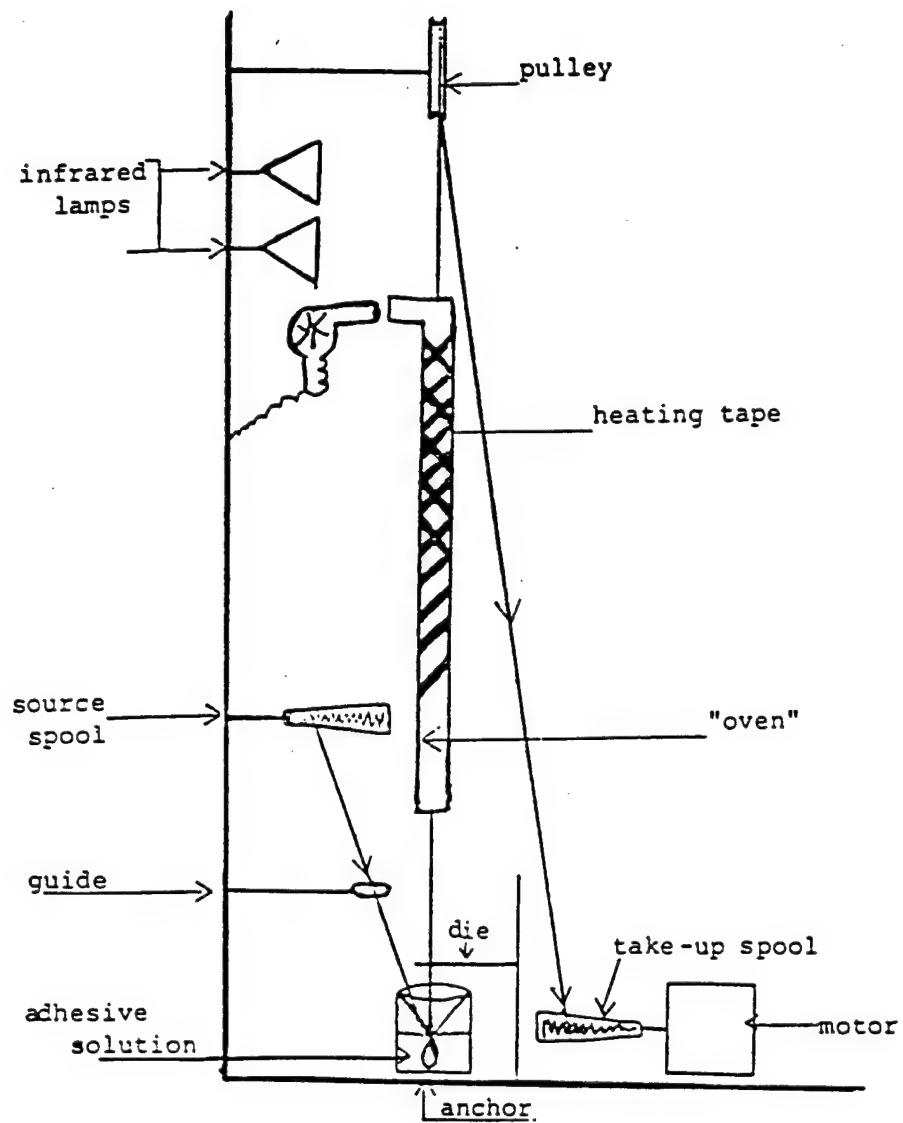


FIGURE 6: Pilot Plant Thread Coater

3. Machine Sewn Samples

Machine sewn samples were prepared using pilot coated green thread, thread which had not been solvent extracted prior to coating. Samples were prepared using thread which had been both double-coated and triple-coated.

In order to compare the effect of sewing process on sealed seam properties, additional samples were also prepared by hand sewing. These samples used a double-coated green thread.

The sewn and sealed samples, which were later subjected to hydrostatic resistance testing, consisted of nylon Taslan/Gore-Tex/tricot knit discs with 6 sewn and sealed stitches. Machine sewn samples were prepared with a standard 301 Lockstitch Singer sewing machine.

The glossy finish on the coated thread became dull after sewing, suggesting a possible loss of some of the coating. The machine sewing created larger needle holes in the fabric than did the hand sewing, which could influence hydrostatic resistance.

Hydrostatic resistance was measured on all samples using a pressure filter. Hydrostatic resistance for both the hand and machine sewn samples was in the range of 12 to 14 psig. This contrasts with the hydrostatic resistance of greater than 35 psig obtained with samples hand sewn and sealed with coated white thread. The white thread, of course, had been solvent extracted prior to coating.

Dye testing was not helpful in determining the leakage path for tested samples.

These results suggest that solvent extraction will be required on all thread prior to coating in order to obtain adequate hydrostatic resistance in the final sewn and sealed seams.

IV. CONCLUSIONS

This Phase I development program successfully demonstrated the concept of a heat sealable sewing thread for use in the outer shell fabric laminate of a chemical protective garment. Specifically, this development effort demonstrated the feasibility of coating a sewing thread with a hot melt resin, using that coated thread to sew a nylon Taslan/Gore-Tex/tricot knit laminate, and then heating the sewn area to produce a seam with hydrostatic resistance of at least 35 psig. The brief 350°F 30-minute heating did not appear to damage the cloth and thread.

Based on this Phase I work, it is feasible to machine sew the coated thread. Volatile fluorocarbon oils have been identified which can be used as thread lubricants to facilitate the machine sewing process.

Successful bonding of nylon thread to Gore-Tex was achieved with a number of hot melts; Elvamide 8061, Nucrel 925, Michem 4990, Estane 5715, TPX 11-808, Elvax 4310, and Macromelts 6301 and 6071. Of the hot melts evaluated for adhesion to polyester and Gore-Tex, the Vitel 4709A showed the most promise.

Michelman/Dow Michem 4990, a waterborne ethylene acrylic acid, was the most promising resin for thread coating. Fabric samples, sewn and sealed with Michem 4990 coated thread, withstood the highest hydrostatic resistance in the most reproducible manner. Using a household sewing machine, thread triple-coated with Michem 4990, sewed with comparable speed to that of the uncoated thread.

V. RECOMMENDATIONS

Sewability

- o The primary consideration for the next phase of this program would be to design a process in which the sewing and heat sealing of the coated thread could be carried out at one work station. The sewing machine station may be adapted in the following ways to incorporate the heat sealing process:

- . two heated 5/8" Teflon coated opposing rollers,
- . one heated roller and a Teflon-coated plate,
- . hot air jets,
- . ultrasonic horn-driven wheel or 1x5/8-inch surface.

- o In Phase I a lubricant was used to aid the sewing process. It was assumed the friction and heat generated during sewing may cause the coating to soften and become tacky, which would in turn impede the sewing. The lubricant (3M's FC-70) is expensive. The following alternatives should be considered:

- . evaluate Teflon-coated needles to reduce friction,
- . evaluate other less expensive lubricants as an alternative lubricant,
- . test various coated threads without lubrication.

- o The two industrial machines (301, 401) presently used produce two different kinds of stitches.

The 301 gives the conventional stitch that looks the same on the needle side as it does on the bobbin side with the interlocking loop occurring (ideally) within the cloth.

The 401 produces the conventional stitch on top, but the underside is a series of loops (a greater thread mass). The 401 is also a faster machine than the 301.

Future work involving these two machines should include the following:

- . Compare the sewability of the two machines with the coated thread. There may only be a need to use coated thread on the bottom stitch due to the increased thread mass and therefore, coating mass.
- . Investigate a steam press for sealing the thread holes in Chemical Protective (CP) uniforms.
- . Investigate methods for factory and field repair of leakers. Develop a liquid formulation.

Formulations

The present study (Phase I) has primarily dealt with nylon and Gore-Tex as the substrates of interest. Ideally, an adhesive (coating) with more universal application is needed. Steps to achieve this should include the following:

- o Future formulations would include new materials and reformulations of old materials (for nylon thread) in order to obtain minimum coating weight for foolproof sealing versus maximum coating weight for sewability (first with household, then industrial sewing machines).
- o Investigate coatings for thread to be used with neoprene-coated waterproof fabric.
- o Research coatings for polyester core thread with spun cotton coating.
- o Evaluate coatings for thread used in vinyl coated polyester (e.g., tents).
- o Determine materials to coat Belding Corticelli's "NYMO" Monocord. Meet with their personnel to discuss substituting our Michem 4990 type binder for their present binder in experimental lots.
- o Investigate ultraviolet resistance with and without UV screening agents in the formulation.
- o Examine formulations and materials for bonding to other barrier resins such as solid TPU sheet (e.g., BION II).
- o Detailed work with foaming formulations should, also, be carried out, especially with foam stabilizers; lesser amounts of foaming agent.
- o Alternate source of supply for Michem 4990.
- o Some work will have to be undertaken to establish whether or not unsized thread has to be utilized for thread coating.
- o It will have to be established if sizes can be utilized on thread for Extended Cold Weather Clothing System (ECWCS) or Aircrew Uniform Integrated Battlefield (AUIB) applications.

Testing

The Phase II program should involve extensive testing of the materials and final products along with failure analysis. The following is suggested:

- o Evaluation of coated thread in ECWCS laminates for water repellant use.
- o Evaluation of coated thread in AUIB laminates for CP use.
- o Investigation of laundering, dry cleaning resistance.
- o Determine thread tensile strength before and after coating with Michem 4990.
- o Evaluation of abrasion resistance of the thread with and without Michem 4990 impregnation.
- o Evaluation of tentage with treated thread by cup test.
- o Investigation of dye leaks with a microscope and/or an electron microscope. Analyze failures, i.e., wicking along the inside of the twisted thread, wicking between thread and Gore-Tex, or possible "trap-door" failure of Gore-Tex.
- o Analyze failure of foam formulations.
- o Evaluate wear and tear testing of the hot melt thread sealed clothing, 5 uniforms.
- o Evaluate flammability.
- o Evaluate agent resistance.

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APPENDIX

Hot Melt Materials

APPENDIX

Hot Melt Materials

| Materials | Manufacturer | Type Resin |
|-------------------|--------------------|---|
| Vitel 4709A | Goodyear | Polyester |
| Vitel 10,202 | Goodyear | Polyester |
| TPX 11-808 | Henkel Corporation | Polyamide |
| Duraflex 8910 | Shell Chemical Co. | Polybutylene |
| Nucrel 699 | DuPont | Ethylene/Methacrylic Acid Copolymer |
| Nucrel 925 | DuPont | Ethylene/Methacrylic Acid Copolymer |
| EAA 459 | Dow | Ethylene/Acrylic Acid |
| Surlyn 9970 | DuPont | Ionomer |
| Estane 5715 | B.F. Goodrich | Polyurethane |
| Estane 5701 | B.F. Goodrich | Polyurethane |
| Bostik 7376 | Bostik | Polyester |
| Bostik 7178 | Bostik | Polyester |
| Bostik 7155 | Bostik | Polyester |
| Michem 4983 Prime | Michelman/Dow | Ethylene Acrylic Acid Water Dispersion |
| Michem 4990 Prime | Michelman/Dow | Ethylene Acrylic Acid Water Dispersion ("Primacor") |
| Elvax 4310 | DuPont | Ethylene Vinyl Acetate Copolymer |
| Adcote 50T4983 | Morton Thiokol/Dow | Ethylene Acrylic Acid Water Dispersion |

| <u>Materials</u> | <u>Manufacturer</u> | <u>Type Resin</u> |
|------------------|---------------------|---|
| Adcote 50T4990 | Morton Thiokol/Dow | Ethylene Acrylic Acid Water Dispersion |
| Hytrel 4056 | DuPont | Polyester |
| NeoRez R-960 | Polyvinyl Chem | Aliphatic Urethane Dispersion |
| Serfene 2060 | Morton Thiokol | Vinylidene Chloride Dispersion |
| Elvamide 8023 | DuPont | Polyamide |
| Elvamide 8061 | DuPont | Polyamide |
| Elvamide 8063 | DuPont | Polyamide |
| Serfene 181 | Morton Thiokol | Vinylidene Chloride Dispersion |
| Macromelt 6071 | Henkel Corporation | Polyamide |
| Macromelt 6301 | Henkel Corporation | Polyamide |
| Uni-Rez 2623 | Union Camp | Polyamide |
| Uni-Rez 2620 | Union Camp | Polymide |
| Uni-Rez 2655 | Union Camp | Polyamide |
| Uni-Rez 2645 | Union Camp | Polyamide |
| Uni-Rez 2665 | Union Camp | Polyamide |
| Versalon XR1165 | Henkel Corporation | Polyamide |
| Versalon 1140 | Henkel Corporation | Polyamide |
| Macromelt 6030 | Henkel Corporation | Polyamide |